



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2585

Organic Contaminants in House Dust

This Standard Reference Material (SRM) is a house dust intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and polybrominated diphenyl ether (PBDE) congeners in house dust and similar matrices. All of the constituents for which certified, reference, and information values are provided in SRM 2585 were naturally present in the dust material before processing. A unit of SRM 2585 consists of one bottle containing approximately 10 g of dust.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 33 PAHs, 30 PCB congeners, 4 chlorinated pesticides, and 15 PBDE congeners are provided in Tables 1 through 4. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques while the certified values for the PBDE congeners are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques and results from a collaborating laboratory [1,2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias have been investigated or accounted for by NIST.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 33 additional PAHs, 12 additional PCB congeners, 10 additional chlorinated pesticides, and 3 additional PBDE congeners in Tables 5 through 8. Reference values are noncertified values that are the best estimate of the true value. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

Information Concentration Values: A NIST information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed. Information values for concentrations, expressed as mass fractions, are provided in Table 9 for nine additional PBDE congeners.

Expiration of Certification: The certification of this SRM lot is valid until **01 June 2015**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification of this material was under the leadership of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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Analytical measurements for the certification of SRM 2585 were performed at NIST by J.M. Keller, J.R. Kucklick, B.J. Porter, D.L. Poster, M.M. Schantz, H.M. Stapleton, and S.S. Vander Pol of the NIST Analytical Chemistry Division.

Additional measurements for selected PBDE congeners were performed at the Meteorological Service of Canada, Environment Canada (Toronto, Canada) by T. Harner and M. Shoeib.

The collection of SRM 2585 was under the direction of the Research Triangle Institute and the U.S. Environmental Protection Agency (Research Triangle Park, NC).

The preparation of SRM 2585 was performed by M.P. Cronise, C.N. Fales, and D.G. Friend of the NIST Measurement Services Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

NOTICE AND WARNING TO USERS

Storage: SRM 2585 must be stored in its original bottle at temperatures less than 15 °C to 30 °C away from direct sunlight.

Handling: This material is naturally occurring house dust collected from a number of locations and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

INSTRUCTIONS FOR USE

Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 2585 are reported on a dry-mass basis. The SRM, as received, contains approximately 2.1 % moisture. The dust sample should be dried to a constant mass before weighing for analysis, or if the constituents of interest are volatile, a separate subsample of the dust should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

PREPARATION AND ANALYSIS¹

Sample Collection: The dust used for the preparation SRM 2585 was taken from vacuum cleaner bags collected from homes, cleaning services, motels, and hotels in the states of North Carolina, Maryland, Ohio, New Jersey, Montana, and Wisconsin during 1993 and 1994. The dust used for the preparation of SRM 2585 was collected at the same time and from the same locations as the dust used for the preparation of SRM 2583 *Trace Elements in Indoor Dust, Nominal 90 mg/kg Lead* [3] and SRM 2584 *Trace Elements in Indoor Dust, Nominal 1 % Lead* [4].

Sample Preparation: The vacuum cleaner bags were first sterilized by gamma irradiation, receiving a minimum total dose of 2.5 mrad. For the low lead material, SRM 2583, the contents of each vacuum cleaner bag was passed through a coarse screen (2 mm hole size) to remove cotton and debris. Then using a vibrating stainless steel sieve apparatus, the resultant material was screened in two successive steps, first through a 250 µm sieve and then a 100 µm sieve. For the high lead material, SRM 2584, the raw material from each bag was mixed and tumbled in a modified food processor using chopping blades and a compressed air jet. While still tumbling, the dust was separated from unwanted debris by vacuuming through a series of screens into a clean HEPA vacuum cleaner. The dust collected in this manner was then screened through a 90 µm stainless steel sieve using vibration and a vacuum. Processed sublots of approximately 5 kg each were set aside and analyzed for lead by X-ray fluorescence to develop a blending protocol for the target lead concentrations in SRM 2584. SRM 2585 was prepared from approximately 70 % of the material found to have 90 mg/kg nominal lead levels used for SRM 2583 and 30 % of the material that had the high lead content used for SRM 2584. The selected sublots were blended in a cone blender and then bottled (approximately 10 g of dust per bottle) as SRM 2585.

Conversion to Dry-Mass Basis: The moisture content of the dust was determined by measuring the mass loss after freeze drying. Six bottles of SRM 2585 were selected according to a stratified randomization scheme for the drying

¹ Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

study. Approximately 1 g of each glass bottle was transferred to a glass weighing vial and dried for seven days at 1 Pa with 5 °C shelf temperature and a -46.9 °C condenser temperature. The moisture content in SRM 2585 at the time of the certification analyses was 2.11 % \pm 0.06 % (95 % confidence level). Analytical results for the organic constituents were determined on an as received mass fraction basis and then converted to a dry-mass fraction basis by dividing by the conversion factor of 0.9789 (g dry mass/g as received mass).

Polycyclic Aromatic Hydrocarbons

The general approach used for the value assignment of the PAHs in SRM 2585 was similar to that reported for the recent certification of several environmental matrix SRMs [5–8] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. The methods utilized Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts by solid phase extraction (SPE), and analysis by gas chromatography/mass spectrometry (GC/MS) of the PAH fraction on three stationary phases of different selectivity, i.e., a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a relatively non-polar proprietary phase, and a liquid crystalline phase.

Seven sets of GC/MS results, designated as GC/MS (Ia), (Ib), (IIa), (IIb), (IIIa), (IIIb), and (IV) were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I and II) analyses, one subsample of approximately 1 g from 6 bottles of SRM 2585 was extracted using PFE with dichloromethane (DCM) [9]. The concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane (volume fraction). The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. \times 60 m fused silica capillary column with 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness) (DB-17MS, J&W Scientific, Folsom, CA). This method is designated as GC/MS (Ia) and GC/MS (IIa). For GC/MS (Ib) and GC/MS (IIb), the same extracts were analyzed by GC/MS using a 0.25 mm i.d. \times 15 m fused silica capillary column with dimethyl 50 % (mole fraction) liquid crystalline polysiloxane phase (0.25 μ m film thickness) (LC-50, J&K Environmental, Sydney, Nova Scotia, Canada). The GC/MS (III) analyses were performed using duplicate subsamples of approximately 1 g from six bottles of SRM 2585. These samples were Soxhlet extracted for 18 h with 250 mL of 50 % hexane and 50 % acetone (volume fraction). The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The fractions were concentrated, and the SPE step was repeated. GC/MS analysis was performed using 0.25 mm i.d. \times 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 μ m film thickness) (DB-XLB, J&W Scientific, Folsom, CA) for method GC/MS (IIIa) and a 50 % phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness) (DB-17MS) for method GC/MS (IIIb). For the GC/MS (IV) analyses, approximately 1 g subsamples from six bottles of SRM 2585 were extracted using PFE with dichloromethane (DCM). The concentrated extract was passed through an alumina cartridge and eluted with 9 mL of 35 % dichloromethane in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. \times 60 m fused silica capillary column with 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness) (DB-17MS). For the GC/MS measurements described above, selected perdeuterated PAHs were added to the dust prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs

The homogeneity of SRM 2585 was assessed by analyzing duplicate samples of approximately 1 g from six bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (IIIa and IIIb). No statistically significant differences among bottles were observed for the PAHs at this sample size.

PCBs and Chlorinated Pesticides

The general approach used for the determination of PCBs and chlorinated pesticides in SRM 2585 was similar to that reported for the recent certification of several environmental matrix SRMs [5,7,10–12], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, cleanup/isolation using normal-phase liquid chromatography (LC) or SPE followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on three columns with different selectivity for the separation of PCBs and chlorinated pesticides.

Four sets of results were obtained, designated as GC-ECD (I), GC/MS (I), GC/MS (II), and GC/MS (III). For GC-ECD (I) and GC/MS (I), subsamples of approximately 1 g from six bottles of SRM 2585 were extracted using PFE with DCM. The concentrated extract was fractionated by LC on a semi-preparative aminopropylsilane column to isolate two fractions containing (1) the PCB congeners and lower polarity pesticides using hexane as the

mobile phase and (2) the more polar pesticides using 25 % DCM in hexane as the mobile phase. Each concentrated fraction was passed through a silica SPE cartridge with 25 % DCM in hexane, concentrated, and then analyzed by GC-ECD and by GC/MS both using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness) (DB-XLB). PCB 30, PCB 103, and perdeuterated 4,4'-DDT were added prior to extraction for use as internal standards for quantification purposes. For GC/MS (II), duplicate subsamples of approximately 1 g from six bottles of SRM 2585 were Soxhlet extracted for 18 h with 250 mL of 50 % hexane and 50 % acetone (volume fraction). The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The fractions were concentrated, and the SPE step was repeated. GC/MS analysis was performed using 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17 MS). For the GC/MS (II) analyses, selected ¹³C labeled PCB congeners and chlorinated pesticides were added to the dust prior to extraction for use as internal standards for quantification purposes. For the GC/MS (III) analyses, approximately 1 g subsamples from six bottles of SRM 2585 were extracted using PFE with dichloromethane (DCM). The concentrated extract was passed through an alumina cartridge and eluted with 9 mL of 35 % dichloromethane in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5MS, J&W Scientific, Folsom, CA). Electron impact mass spectrometry was used for all of the analytes except for the chlordanes and nonachlors, which were quantified using negative chemical ionization for GC/MS (III). For the GC/MS (III) analyses, selected ¹³C labeled PCB congeners, ¹³C labeled *trans*-chlordane, and selected deuterated pesticides were added to the dust prior to extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PCBs and Chlorinated Pesticides: The homogeneity of SRM 2585 was assessed by analyzing duplicate samples of approximately 1 g from six bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (II). No statistically significant differences among bottles were observed for the chlorinated analytes at this sample size.

Polybrominated Diphenyl Ethers

Value assignment of concentrations for PBDE congeners was based on four sets of measurements (three sets from NIST and one set from a collaborating laboratory) using a variety of different extraction, cleanup, and quantification methods. All measurements were performed by using GC/MS operated in either electron impact (GC/EI-MS) or negative chemical ionization (GC/NCI-MS) mode.

For two of the NIST measurement sets, GC/NCI-MS (I) and GC/EI-MS(I), between 0.5 g and 1 g subsamples of dust from each of four bottles were extracted using PFE with DCM. The concentrated extract was placed on a silica SPE cartridge and eluted with hexane. The extracts were analyzed by using both GC/EI-MS and GC/NCI-MS on a 0.25 mm × 15 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W Scientific, Folsom, CA). For both methods ¹³C-labeled 2,2',3,4,5-pentachlorodiphenyl ether (CDE 86L) and ¹³C-labeled 2,2',3,3',4,4',5,5',6,6'-decabromodiphenyl ether (BDE 209L) were added to the dust sample prior to extraction for use as internal standards for quantification purposes.

For the third NIST measurement set, GC/EI-MS(II), between 0.5 g and 1 g subsamples of dust from each of six bottles were extracted using PFE with DCM. The extracts were first subjected to a cleanup step using a 5 % deactivated alumina SPE column and then injected onto a size exclusion chromatographic column. The extracts were analyzed by using GC/EI-MS on a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-5MS, J&W Scientific, Folsom, CA). ¹³C-labeled 2,2,4,4',5-pentabromodiphenyl ether (PBDE 99L) and PBDE 209L were added to the dust samples prior to extraction for use as an internal standard for quantification of the PBDEs.

For the measurements from the collaborating laboratory (Environment Canada), GC/NCI-MS(II), four subsamples of between 0.5 g and 1 g from a single bottle were Soxhlet extracted using DCM after spiking with mirex as an internal standard. The extracts were cleaned using 2 g of 6 % deactivated alumina with petroleum ether. The extracts were analyzed by using GC/NCI-MS on a 0.25 mm × 15 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-5).

Table 1. Certified Concentrations for Selected PAHs in SRM 2585

	Mass Fraction (dry-mass basis) ^(a) μg/kg		
Naphthalene ^(d,e,f)	266	±	8 ^(b)
Dibenzothiophene ^(d,e,f,g)	109	±	8 ^(c)
Phenanthrene ^(d,e,f,g,h,i)	1920	±	20 ^(c)
Anthracene ^(d,e,f,g,h,i)	96.0	±	5.2 ^(c)
4H-cyclopenta[<i>def</i>]phenanthrene ^(d,e,f,g)	117	±	10 ^(c)
3-Methylphenanthrene ^(e,f,g,i)	293	±	36 ^(c)
2-Methylphenanthrene ^(e,f,g)	352	±	40 ^(b)
9-Methylphenanthrene ^(e,f,g,i)	205	±	16 ^(c)
1-Methylphenanthrene ^(e,f,g)	197	±	29 ^(b)
Fluoranthene ^(d,e,f,g,h,i)	4380	±	100 ^(c)
Pyrene ^(d,e,f,g,h,i)	3290	±	30 ^(c)
Benzo[<i>ghi</i>]fluoranthene ^(d,e,f,g,h,i)	317	±	11 ^(c)
Benzo[<i>c</i>]phenanthrene ^(d,e,f,g,h,i)	288	±	10 ^(c)
Benzo[<i>a</i>]anthracene ^(d,e,f,g,h,i)	1160	±	54 ^(c)
Chrysene ^(e,h,i)	2260	±	60 ^(b)
Triphenylene ^(e,h,i)	589	±	17 ^(b)
Benzo[<i>b</i>]fluoranthene ^(d,f,g,h,i,j)	2700	±	90 ^(c)
Benzo[<i>j</i>]fluoranthene ^(d,f,g,i)	1320	±	110 ^(c)
Benzo[<i>k</i>]fluoranthene ^(d,f,g,h,j)	1330	±	70 ^(c)
Benzo[<i>a</i>]fluoranthene ^(d,f,g,j)	74.5	±	8.1 ^(c)
Benzo[<i>e</i>]pyrene ^(d,f,g,h,i,j)	2160	±	80 ^(c)
Benzo[<i>a</i>]pyrene ^(d,f,g,h,i,j)	1140	±	10 ^(c)
Perylene ^(d,f,g,h,i,j)	387	±	23 ^(c)
Benzo[<i>ghi</i>]perylene ^(d,f,g,h,i,j)	2280	±	40 ^(c)
Indeno[1,2,3- <i>cd</i>]pyrene ^(d,f,g,h,i,j)	2080	±	100 ^(c)
Dibenz[<i>a,j</i>]anthracene ^(d,f,g,h,i,j)	267	±	9 ^(c)
Dibenz[<i>a,c</i>]anthracene ^(d,f,g,h,i,j)	183	±	25 ^(c)
Dibenz[<i>a,h</i>]anthracene ^(d,f,g,h,i,j)	301	±	50 ^(c)
Benzo[<i>b</i>]chrysene ^(d,f,g,h,i)	182	±	6 ^(c)
Picene ^(d,f,g,h,j)	413	±	15 ^(c)
Coronene ^(d,f,g,h,i,j)	603	±	38 ^(c)
Dibenzo[<i>b,k</i>]fluoranthene ^(d,f,g,h,i,j)	596	±	22 ^(c)
Dibenzo[<i>a,e</i>]pyrene ^(d,f,g,h,i,j)	477	±	67 ^(c)

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

^(b) Certified values are unweighted means of the results from three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO and NIST Guides [2].

^(c) Certified values are weighted means of the results from four to six analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), except for phenanthrene and benzo[*a*]pyrene for which a coverage factor of 4 was used, calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [2].

^(d) GC/MS (Ia) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

^(e) GC/MS (IIIa) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

^(f) GC/MS (IV) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

^(g) GC/MS (IIa) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

^(h) GC/MS (Ib) on a liquid crystalline phase after PFE with DCM.

⁽ⁱ⁾ GC/MS (IIb) on a liquid crystalline phase after PFE with DCM.

^(j) GC/MS (IIIb) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 2. Certified Concentrations for Selected PCB Congeners in SRM 2585

PCB Congener ^(a)		Mass Fraction (dry-mass basis) ^(b) µg/kg		
PCB 18	(2,2',5-Trichlorobiphenyl) ^(e,f,g,h)	12.8	±	1.0 ^(c)
PCB 28	(2,4,4'-Trichlorobiphenyl) ^(f,g)	13.4	±	0.5 ^(d)
PCB 31	(2,4',5-Trichlorobiphenyl) ^(f,g)	14.0	±	0.5 ^(d)
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl) ^(e,f,g,h)	18.1	±	1.9 ^(c)
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl) ^(e,f,g,h)	21.8	±	1.9 ^(c)
PCB 56	(2,3,3',4-Tetrachlorobiphenyl) ^(e,f,g)	4.42	±	0.28 ^(d)
PCB 70	(2,3',4',5-Tetrachlorobiphenyl) ^(e,f,g,h)	13.1	±	1.2 ^(c)
PCB 74	(2,4,4',5-Tetrachlorobiphenyl) ^(e,f,g,h)	5.22	±	0.51 ^(c)
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl) ^(e,f,g,h)	16.6	±	0.8 ^(c)
PCB 92	(2,2',3,5,5'-Pentachlorobiphenyl) ^(f,g,h)	5.48	±	0.72 ^(d)
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl) ^(f,g,h)	22.7	±	2.6 ^(d)
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl) ^(f,g,h)	11.6	±	0.4 ^(c)
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl) ^(f,g,h)	29.8	±	2.3 ^(d)
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl) ^(f,g,h)	13.2	±	1.4 ^(d)
PCB 107	(2,3,3',4,5'-Pentachlorobiphenyl) ^(e,f,g)	4.14	±	0.47 ^(d)
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl) ^(e,f,g,h)	28.1	±	3.7 ^(c)
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl) ^(e,f,g,h)	26.3	±	1.7 ^(c)
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl) ^(e,f,g,h)	27.6	±	2.1 ^(c)
PCB 146	(2,2',3,4',5,5'-Hexachlorobiphenyl) ^(e,f,g,h)	4.89	±	0.38 ^(c)
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl) ^(e,f,g,h)	24.4	±	1.9 ^(c)
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl) ^(f,g,h)	6.92	±	0.64 ^(d)
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl) ^(e,f,g,h)	40.2	±	1.8 ^(c)
132	(2,2',3,3',4,6'-Hexachlorobiphenyl) ⁽ⁱ⁾			
PCB 158	(2,3,3',4,4',6-Hexachlorobiphenyl) ^(e,f,g,h)	4.50	±	0.43 ^(c)
PCB 163	(2,3,3',4',5,6-Hexachlorobiphenyl) ^(e,f,g,h)	7.2	±	1.2 ^(c)
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl) ^(e,f,g,h)	8.8	±	1.0 ^(c)
PCB 174	(2,2',3,3',4,5,6'-Heptachlorobiphenyl) ^(e,f,g,h)	8.83	±	0.47 ^(c)
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) ^(e,f,h)	18.4	±	3.2 ^(d)
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl) ^(e,f,g,h)	5.27	±	0.39 ^(c)
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl) ^(e,f,g,h)	11.3	±	1.4 ^(c)
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) ^(e,h)	3.81	±	0.13 ^(d)

^(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [15] and later revised by Schulte and Malisch [16] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, only PCB 107 and PCB 201 (see Table 6) are different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 107 is listed as PCB 108 and the IUPAC PCB 201 is listed as PCB 200.

^(b) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

^(c) Certified values are weighted means of the results from four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [2].

^(d) Certified values are unweighted means of the results from two or three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO and NIST Guides [2].

^(e) GC-ECD (I) on a relatively non-polar proprietary phase after PFE extraction with DCM.

^(f) GC/MS (I) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (I).

^(g) GC/MS (II) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

^(h) GC/MS (III) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

⁽ⁱ⁾ PCB 153 is the primary component with PCB 132 contributing at most 10 % to the concentration based on the data from method GC/MS (II).

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 2585

	Mass Fraction (dry-mass basis) ^(a) µg/kg		
4,4'-DDE ^(d,e,f,g)	261	±	2 ^(b)
4,4'-DDD ^(d,f)	27.3	±	0.8 ^(c)
2,4'-DDT ^(d,f)	44.5	±	3.9 ^(c)
4,4'-DDT ^(d,f,g)	111	±	23 ^(c)

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

^(b) Certified values are weighted means of the results from four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) except for 4,4'-DDE with a coverage factor of 10, calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [2].

^(c) Certified values are unweighted means of the results from two or three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO and NIST Guides [2].

^(d) GC-ECD (I) on a relatively non-polar proprietary phase after PFE extraction with DCM.

^(e) GC/MS (I) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (I).

^(f) GC/MS (II) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

^(g) GC/MS (III) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

Table 4. Certified Concentrations for Selected PBDE Congeners in SRM 2585

PBDE Congener ^(a)		Mass Fraction (dry-mass basis) µg/kg		
PBDE 17	(2,2',4-Tribromodiphenyl Ether) ^(c,d,e,f)	11.5	±	1.2 ^(b)
PBDE 28 ^(g)	(2,4,4'-Tribromodiphenyl Ether) ^(c,d,e,f)	46.9	±	4.4 ^(b)
PBDE 33	(2',3,4-Tribromodiphenyl Ether)			
PBDE 47	(2,2',4,4'-Tetrabromodiphenyl Ether) ^(c,d,e,f)	497	±	46 ^(b)
PBDE 49	(2,2',4,5'-Tetrabromodiphenyl Ether) ^(c,d,e)	53.5	±	4.2 ^(h)
PBDE 85	(2,2',3,4,4'-Pentabromodiphenyl Ether) ^(c,d,e,f)	43.8	±	1.6 ^(b)
PBDE 99	(2,2',4,4',5-Pentabromodiphenyl Ether) ^(c,d,e,f)	892	±	53 ^(b)
PBDE 100	(2,2',4,4',6-Pentabromodiphenyl Ether) ^(c,d,e,f)	145	±	11 ^(b)
PBDE 138	(2,2',3,4,4',5'-Hexabromodiphenyl Ether) ^(c,d,e,f)	15.2	±	2.0 ^(b)
PBDE 153	(2,2',4,4',5,5'-Hexabromodiphenyl Ether) ^(c,d,e,f)	119	±	1 ^(b)
PBDE 154	(2,2',4,4',5,6'-Hexabromodiphenyl Ether) ^(d,e)	83.5	±	2.0 ^(h)
PBDE 155	(2,2',4,4',6,6'-Hexabromodiphenyl Ether) ^(d,e)	3.94	±	0.34 ^(h)
PBDE 183	(2,2',3,4,4',5',6-Heptabromodiphenyl Ether) ^(c,d,e,f)	43.0	±	3.5 ^(b)
PBDE 203	(2,2',3,4,4',5,6',6-Octabromodiphenyl Ether) ^(c,e)	36.7	±	6.4 ^(h)
PBDE 206	(2,2',3,3',4,4',5,5',6-Nonabromodiphenyl Ether) ^(c,e)	271	±	42 ^(h)
PBDE 209	(Decabromodiphenyl Ether) ^(c,e,f)	2510	±	190 ^(h)

^(a) PBDE congeners are numbered according to IUPAC rules.

^(b) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture. Certified values are weighted means of the results from four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) except for PBDE 153 with a coverage factor of 10, calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [2].

^(c) GC/NCI-MS (I) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

^(d) GC/EI-MS (I) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM (same extracts as GC/NCI-MS(I)).

^(e) GC/EI-MS (II) on a 60 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

^(f) GC/NCI-MS (II) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^(g) Using the 15 m column, PBDE 28 and 33 coelute; however, using the 60 m column, the two isomers are separated. Based on the data from the 60 m column, the concentration of PBDE 33 is less than the detection limit of 2.2 µg/kg.

^(h) Certified values are unweighted means of the results from two or three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [23] with a pooled, within-method variance following the ISO and NIST Guides [2].

Table 5. Reference Concentrations for Selected PAHs in SRM 2585

	Mass Fraction (dry-mass basis) ^(a) µg/kg		
1-Methylnaphthalene ^(d,e,f)	150	±	37 ^(b)
2-Methylnaphthalene ^(d,e,f)	227	±	20 ^(b)
Biphenyl ^(e,f,g)	88	±	21 ^(b)
Retene ^(d)	588	±	34 ^(c)
1,7-Dimethylphenanthrene ^(f,g)	219	±	19 ^(b)
1-Methylfluoranthene ^(d,f,h)	94	±	11 ^(b)
3-Methylfluoranthene ^(d,f,h)	235	±	67 ^(b)
8-Methylfluoranthene ^(d)	132	±	7 ^(c)
4-Methylpyrene ^(d,f,h)	235	±	27 ^(b)
2-Methylpyrene ^(d)	345	±	10 ^(c)
1-Methylpyrene ^(d,f,h)	209	±	69 ^(b)
3-Methylchrysene ^(d,f,h)	146	±	18 ^(b)
2-Methylchrysene ^(d)	181	±	4 ^(c)
6-Methylchrysene ^(d,f)	88	±	14 ^(b)
4-Methyl- and 1-Methylchrysene ^(d)	94.8	±	4.8 ^(c)
9-Methyl- and 3-Methylbenz[<i>a</i>]anthracene ^(d)	92.3	±	2.5 ^(c)
6-Methyl- and 1-Methylbenz[<i>a</i>]anthracene ^(d)	155	±	5 ^(c)
Anthanthrene ^(f,g,i)	91	±	27 ^(b)
Dibenzo[<i>b,e</i>]fluoranthene ^(f,g,j)	59.6	±	7.5 ^(b)
Naphtho[1,2- <i>b</i>]fluoranthene ^(g,i)	312	±	10 ^(b)
Naphtho[1,2- <i>k</i>] and naphtho[2,3- <i>j</i>]fluoranthene ^(f,g,j)	382	±	18 ^(b)
Naphtho[2,3- <i>b</i>]fluoranthene ^(f,g,i)	93	±	30 ^(b)
Naphtho[2,3- <i>k</i>]fluoranthene ⁽ⁱ⁾	24.7	±	1.2 ^(c)
Dibenzo[<i>a,k</i>]fluoranthene ^(f,j)	14.3	±	3.4 ^(b)
Dibenzo[<i>j,l</i>]fluoranthene ^(f,g,i,j)	260	±	26 ^(k)
Dibenzo[<i>a,l</i>]pyrene ^(f,g,j)	42.3	±	3.1 ^(b)
Naphtho[2,3- <i>k</i>]fluoranthene and Naphtho[1,2- <i>a</i>]pyrene ^(f,g,i)	44.3	±	1.9 ^(b)
Naphtho[2,3- <i>e</i>]pyrene ^(f,g,i)	145	±	29 ^(b)
Naphtho[2,1- <i>a</i>]pyrene ^(f,g,i,j)	379	±	47 ^(k)
Dibenzo[<i>e,l</i>]pyrene ^(f,g,i,j)	208	±	14 ^(k)
Benzo[<i>b</i>]perylene ^(g,i)	103	±	24 ^(b)
Dibenzo[<i>a,i</i>]pyrene ^(g,i)	105	±	11 ^(b)
Dibenzo[<i>a,h</i>]pyrene ^(f)	20.9	±	0.7 ^(c)

(a) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

(b) Reference values are unweighted means of the results from two or three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO and NIST Guides [2].

(c) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U , is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k , is determined from the Student's t -distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte.

(d) GC/MS (IIa) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

(e) GC/MS (IIIa) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(f) GC/MS (IV) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

(g) GC/MS (Ia) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

(h) GC/MS (IIb) on a liquid crystalline phase after PFE with DCM.

(i) GC/MS (Ib) on a liquid crystalline phase after PFE with DCM.

(j) GC/MS (IIIb) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(k) Reference values are weighted means of the results from four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [2].

Table 6. Reference Concentrations for Selected PCB Congeners in SRM 2585

PCB Congener ^(a)		Mass Fraction (dry-mass basis) ^(b) µg/kg		
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl) ^(f,g,h,i)	16.4	±	3.3 ^(c)
PCB 63	(2,3,4',5-Tetrachlorobiphenyl) ^(f)	6.69	±	0.26 ^(d)
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl) ^(g,h,i)	8.5	±	1.9 ^(e)
PCB 121	(2,3',4,5',6-Pentachlorobiphenyl) ⁽ⁱ⁾	18.7	±	0.4 ^(d)
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl) ^(h,i)	8.1	±	1.6 ^(e)
PCB 177	(2,2',3,3',4,5,6-Heptachlorobiphenyl) ⁽ⁱ⁾	5.50	±	0.44 ^(d)
PCB 178	(2,2',3,3',5,5',6'-Heptachlorobiphenyl) ⁽ⁱ⁾	2.17	±	0.16 ^(d)
PCB 185	(2,2',3,4,5,5',6-Heptachlorobiphenyl) ⁽ⁱ⁾	5.32	±	0.39 ^(d)
PCB 193	(2,3,3',4',5,5',6-Heptachlorobiphenyl) ^(f)	1.23	±	0.070 ^(d)
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl) ^(f,i)	4.47	±	0.76 ^(e)
PCB 199	(2,2',3,3',4,5,6,6'-Octachlorobiphenyl) ⁽ⁱ⁾	5.81	±	0.38 ^(d)
PCB 209	Decachlorobiphenyl ^(f)	2.14	±	0.11 ^(d)

^(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [15] and later revised by Schulte and Malisch [16] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, only PCB 201 and PCB 107 (see Table 2) are different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 201 is listed as PCB 200 and the IUPAC PCB 107 is listed as PCB 108.

^(b) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

^(c) Reference values are weighted means of the results from four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [2].

^(d) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U , is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k , is determined from the Student's t -distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte.

^(e) Reference values are unweighted means of the results from two to three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO and NIST Guides [2].

^(f) GC-ECD (I) on a relatively non-polar proprietary phase after PFE extraction with DCM.

^(g) GC/MS (I) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (I).

^(h) GC/MS (II) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

⁽ⁱ⁾ GC/MS (III) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

Table 7. Reference Concentrations for Selected Chlorinated Pesticides in SRM 2585

	Mass Fraction (dry-mass basis) ^(a) µg/kg		
<i>cis</i> -Chlordane ^(d,e,f)	174	±	45 ^(b)
<i>trans</i> -Chlordane ^(d,e,f)	277	±	96 ^(b)
<i>cis</i> -Nonachlor ^(f)	28.0	±	0.6 ^(c)
<i>trans</i> -Nonachlor ^(d,e,f)	130	±	38 ^(b)
Heptachlor ^(d,f)	166	±	34 ^(b)
Heptachlor Epoxide ^(f)	11.3	±	0.6 ^(c)
Dieldrin ^(d,f)	88	±	21 ^(b)
<i>gamma</i> -HCH ^(d)	4.06	±	0.55 ^(c)
Mirex ^(d)	6.89	±	0.25 ^(c)
Pentachlorobenzene ^(f)	20.9	±	1.6 ^(c)

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

^(b) Reference values are unweighted means of the results from two to three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO and NIST Guides [2].

^(c) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U , is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k , is determined from the Student's t -distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte.

^(d) GC-ECD (I) on a relatively non-polar proprietary phase after PFE extraction with DCM.

^(e) GC/MS (II) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

^(f) GC/MS (III) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

Table 8. Reference Concentrations for Selected PBDE Congeners in SRM 2585

PBDE Congener ^(a)	Mass Fraction (dry-mass basis) ^(b) µg/kg		
PBDE 66 (2,3',4,4'-Tetrabromodiphenyl ether) ^(d,e,f,g)	29.5	±	6.2 ^(c)
PBDE 75 (2,4,4',6-Tetrabromodiphenylether) ^(e,f)	4.5	±	1.2 ^(h)
PBDE 190 (2,3,3',4,4',5,6-Heptabromodiphenyl ether) ^(d,g)	5.1	±	2.9 ^(h)

^(a) PBDE congeners are numbered according to the IUPAC rules.

^(b) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

^(c) Reference values are weighted means of the results from four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [2].

^(d) GC/NCI-MS (I) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

^(e) GC/EI-MS (I) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM (same extracts as GC/NCI-MS(I)).

^(f) GC/EI-MS (II) on a 60 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

^(g) GC/NCI-MS (II) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^(h) Reference values are unweighted means of the results from two analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO and NIST Guides [2].

Table 9. Information Concentrations for Selected PBDE Congeners in SRM 2585^(a)

PBDE	Congener ^(b)	Mass Fraction (dry-mass basis) ^(c) µg/kg
PBDE 25	(2,3',4-Tribromodiphenyl Ether)	< 0.2
PBDE 30	(2,4,6-Tribromodiphenyl Ether)	< 0.2
PBDE 71	(2,3',4',6-Tetrabromodiphenyl Ether)	< 0.2
PBDE 116	(2,3,4,5,6-Pentabromodiphenyl Ether)	< 0.2
PBDE 119	(2,3',4,4',6-Pentabromodiphenyl Ether)	< 0.2
PBDE 156	(2,3,3',4,4',5-Hexabromodiphenyl Ether)	< 0.2
PBDE 181	(2,2',3,4,4',5,6-Heptabromodiphenyl Ether)	< 0.3
PBDE 191	(2,3,3',4,4',5',6-Heptabromodiphenyl Ether)	< 0.3
PBDE 205	(2,3,3',4,4',5,5',6-Octabromodiphenyl Ether)	< 0.5

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

^(b) PBDE congeners are numbered according to the IUPAC rules.

^(c) The information values are from the method detection limits using a signal to noise value = 100 for method GC/NCI-MS (I).

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